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13. ABSTRACT (Maximum 200 Words)  Research groups at three different universities have worked to develop fundamental, detailed models of thermal and mechanical behavior of carbon foams, and studied surface modification techniques to enable transition of the foam into several aerospace applications. The research team developed a numerical model to predict thermal conductivity, and have identified, by experimentation, reliable techniques to determine the thermal conductivity of carbon foams. The team developed a micromechanics model that includes the effects of cell edges (struts) on the elastic properties of carbon foams; and studied the influence of foam cell size and shape on the macroscopic mechanical behavior of carbon foams using the Voronoi tessellation technique. Team members have also developed surface modification techniques for environmental stability and interface enhancement. The experimental work included enhancement of surface wettability, adhesion/bond control, and coatings for extreme applications.				
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# **Final Report**

**Project Title: Science and Engineering of Carbon Foams**

AFOSR Contract Number F49620-03-1-1004

Submitted by

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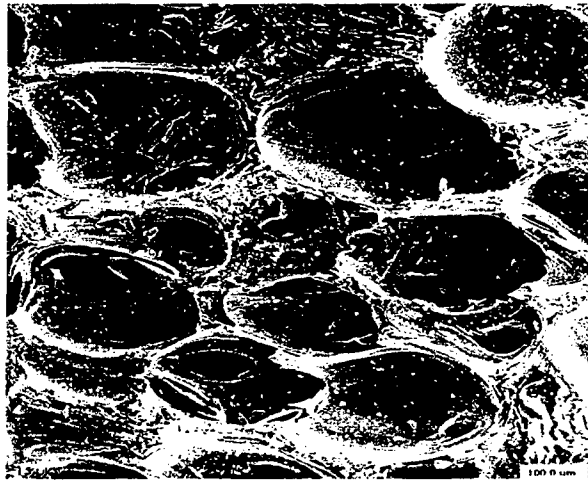
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## 1.0 Executive Summary

Carbon foam is a new class of lightweight cellular materials that was first developed at the Air Force Research Laboratory (AFRL) in 1990s. Although the foam production process tends to vary, many processes start with a compacted, porous pre-form of pitch material. The pitch pre-form is then melted under high pressure of an inert gas. When the pitch material begins to flow above its softening temperature (typically 250°C), the pressure is released to expand trapped gases in the voids, thereby producing foam from the pitch. The product is then stabilized at about 150°C, and carbonized at about 1000°C. A heat treatment step is often used to graphitize the foam at temperatures over 2000°C. The product has a 3-dimensional morphology with closed or open pores; the porosity varies from 60% to 95%. Typical foam pores range from 50 microns to 500 microns in diameter. Most of these foams tend to have open cell structures. An example of foam with such micro-pores is shown in Figure-1. This is graphitic carbon foam with a relative density of 0.2 (80% porosity). The cells are interconnected, and the open cell structure varies from process to process; therefore, foam of different porosities can be produced.

Several factors influence the properties of carbon foams. The nature and amount of porosity of the foam is a major factor in determination of thermo-mechanical properties. Another factor is the degree of graphitization (or the graphitization index) of the carbon in the foam, which depends on the precursor and heat treatment. Depending on the graphitization index, the thermal conductivity of solid carbon ranges from ~1W/mK to 2000W/mK. The graphitization index of a graphitic carbon is a measure of its degree of perfection. It is defined as the probability of finding two layers of graphene (a single layer of hexagonally-arranged carbon atoms) with the A-B stacking in the crystallographic definition of graphite. Un-graphitized carbon foams exhibit very low thermal conductivities, and are excellent lightweight insulators that can survive high temperatures in a non-oxidizing environment. These foams are candidates for aerospace applications such as structural insulation in exhaust-washed structures. While foams in general tend to be poor conductors and weak in mechanical properties, the carbon foam with high degree of graphitization results in high bulk thermal and electrical conductivity and good mechanical properties. Highly graphitic foams can have bulk thermal conductivity higher

than 150W/mK (Ref. 1) and mechanical strength in excess of 200 psi (Ref. 2). Because of the high porosity and high intrinsic thermal conductivity of the solid phase, the specific thermal conductivity of such foams can be several times greater than copper. Therefore, these foams are good candidates for applications in aerospace thermal management, including heat sinks, heat spreaders, cold plates and heat exchanger cores.



**Figure-1:** Micrograph of graphitic carbon foam

The advantage of carbon foam is that it is a multifunctional material, providing a range of thermal and electrical properties along with structural integrity, low density, dimensional stability, low coefficient of thermal expansion (CTE), and high surface area. Therefore, carbon foam is expected to have the greatest impact where several of these properties are critical to the application. Along with low density and thermal insulation properties, the structural integrity is very important when the foam is to be used in the leading edge of a high speed air vehicle, or the structural backing of a rocket nozzle liner. On the other hand, the open porosity, high surface area and high thermal conductivity properties become highly advantageous when the application is the dissipation of heat from an electronic chassis. In the case of a structural radiator in a spacecraft,

the requirements are good mechanical properties, high thermal conductance, and a low CTE that is compatible with a composite structure.

The thermo-mechanical properties of foam are critical for aerospace applications because the foam must conduct (or insulate) while sustaining service and launch loads, and mechanical stresses that arise due to vibrations. Experimental measurements on foams have shown that the compressive strengths and modulus vary over a wide range due to differences in the precursor, processing, pore geometry and density. While the strength is expected to change with density, the strength has also been observed to change from 200 psi to 2000 psi due to processing differences (Ref. 2). The wide range of thermal and mechanical properties makes theoretical and experimental prediction of foam properties an important and complex task; this is compounded by the fact that reliable measurement of thermal properties of foams can be quite difficult. For example, the reliability of using the laser flash technique for thermal diffusivity measurement of a foam specimen is subject to controversy. Because of uncertainties in thermal modeling and experimental characterization of thermal properties of foams, a combination of theoretical and numerical analyses must be combined with experimental validation.

It should be noted that energy transport and stress transfer occur by fundamentally different mechanisms. Therefore, two different geometrical models incorporating variation of density and microstructure will be developed in this study to focus on the thermal and mechanical properties. The thermal model focuses on reproducing the heat conduction path whereas the mechanical model emphasizes randomness of the mechanical structure.

The mechanical structure-property relations are strongly dependent on the randomness in their microstructures, and, consequently, their structure-property relations cannot be completely described by using a traditional micromechanical model alone. Statistical models that allow variations of density and microstructure are therefore needed to complement micromechanical modeling. The similarities between the Voronoi tessellation of space and many carbon foams make the random Voronoi technique an ideal statistical tool for modeling microstructures of such carbon foams.

The issue of foam surface properties presents a special processing challenge for several applications. The high surface area can be effective in thermal transport and in stress transfer when the foam is infiltrated to produce a composite. However, the graphitic surface resists wetting and bonding, and therefore interface adhesion can be a problem. The large surface area can also lead to high oxidation rates at high temperatures. In applications such as heat shield or heat exchangers, the oxidation issue is the limiting factor. Therefore the environmental stability and interface properties need to be improved for specific applications. The surface modification processes can include coatings for oxidation protection or interface bonding, and plasma treatment to improve wetting.

Research groups at three different universities have worked cooperatively in addressing these critical tasks. These tasks and the investigators from the universities are shown below:

**(A) Thermal Transport in Carbon Foams**

Investigator - M. K. Alam, Ph.D., P.E., Principal Investigator, Department of Mechanical Engineering, Ohio University, Athens OH 45701

**(B) Micromechanical modeling of carbon foam**

Investigator: Dr. Xin-Lin Gao, Department of Mechanical Engineering, Texas A & M University, College Station, TX 77843

**(C) Coatings for Surface Modification of Carbon Structures**

Investigator: Dr. Sharmila Mukhopadhyay, Mechanical and Materials Engineering Department, Wright State University, Dayton, OH 45434

The research results are summarized below for each of the three tasks.

## 1.1 Task (A): Thermal Transport in carbon Foams

### 1.11 Theory for thermal transport

Carbon foam can be modeled in the manner of a porous media. Many of these models are based on the analysis of packed beds (Ref. 3-5). All these models have as starting point the following relation:

$$R = \frac{\rho_b}{\rho_s} = 1 - \frac{P}{100} \quad (1)$$

Where,  $\rho_b$  is the bulk foam density,  $\rho_s$  is the intrinsic density of the solid,  $P$  is the porosity of the porous material, and  $R$  is the relative density. The value of thermal conductivity of foam with non-conducting pores has a limiting value given by:

$$K_b \leq K_s \left( \frac{\rho_b}{\rho_s} \right) \quad (2)$$

Where,  $K_b$  is the bulk thermal conductivity, and  $K_s$  is the solid (intrinsic) thermal conductivity. The maximum value is obtained when the porous solid presents the minimum resistance to heat flow; this is given by the rule of mixtures. In practice however, the flow path is tortuous, and the true value is always less than the maximum predicted by Eq. (2). A more rigorous approach was adopted by Bauer (Ref. 6) who considered the perturbation of a continuous medium by the presence of pores. The analytical solution, obtained from the perturbation of a uniform temperature gradient in the  $z$  direction in a uniform medium due to the presence of a spherical pore for solid and bulk thermal conductivity much higher than pore (gas) conductivity (applicable to carbon foams) can be expressed as:

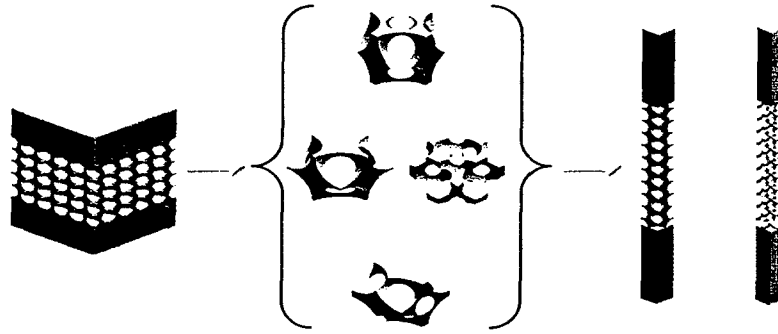
$$\frac{K_b}{K_s} = K_e = \left( 1 - \frac{P}{100} \right)^{1/n} = R^{1/n} \quad (3)$$



Where,  $K_e$  is the effective thermal conductivity of the porous medium. It is important to note that the pore conduction parameter  $n$  has to be determined experimentally for different pore shapes and concentrations. In this study, we use finite element method to analyze the influence of the pore size, pore shape and pore distribution on the effective thermal conductivity of porous materials.

### 1.12 Finite Element Model

To study the influence of the pore shape and distribution, several models have been built using solid modeling software. Examples of these models are shown in Figure-2.



**Figure-2:** Foam solid model, unit cell and finite element model

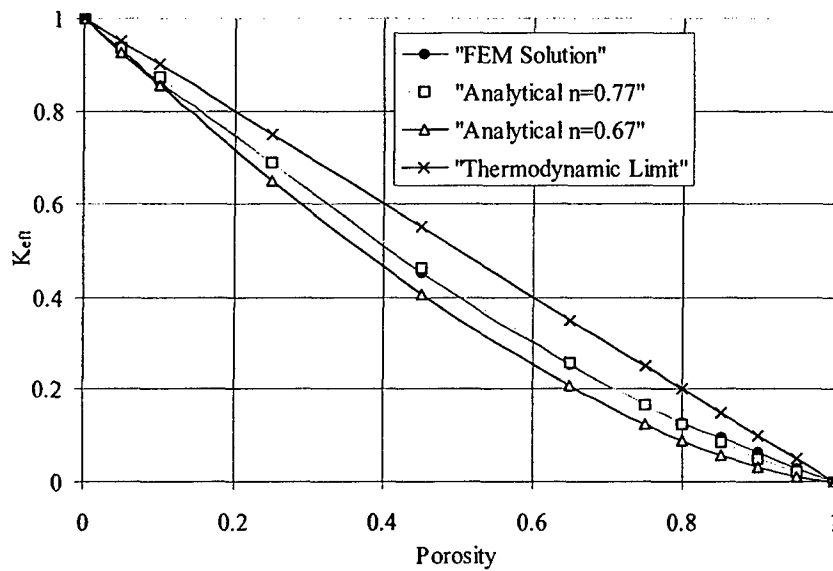
The finite element method is based on the Fourier's law of heat conduction governs the steady state heat transfer through a solid. Over the entire three-dimensional volume domain ( $V$ ), for an isotropic material, the heat conduction without internal heat generation can be described by the following integral formulation (Ref. 7):

$$\iint_V \vec{\nabla} \cdot \vec{q} d\Omega = \iint_V \nabla \cdot (K \nabla T) dV = 0 \quad (4)$$

Where,  $\vec{q}$  is the heat flux vector, and  $K$  is the thermal conductivity and  $T$  is the temperature.

### 1.13 Influence of Pore Volume Concentration

The basic model chosen to study the spherical and elliptical pore foams consisted of a volume centered (VC) cubical model. The pore volume concentration or the porosity of the foam is expected to be a dominant parameter in the thermal conductivity of the foam. Higher porosity foams have less solid for conduction and therefore the conductivity will be reduced. Figure-3 shows the effective thermal conductivity ( $K_e$ ) for foam with body centered pore distribution.



**Figure-3:** Effective thermal conductivity as a function of porosity

Using Eq. (3) proposed by Bauer (Ref. 6), the conduction parameter  $n$  can be found using the equation:

$$n = \frac{\ln R}{\ln K_e} = \frac{\ln(1 - P/100)}{\ln K_e} \quad (5)$$

Using the finite element results, the conduction parameter can be determined for a wide range of porosities.

#### 1.14 Influence of Pore Distribution on the Thermal Conductivity

In order to study the influence of the arrangement of the pores on the transport properties of the foam, a different pore arrangement was also analyzed. The model considered (presented in Figure 2) is a face centered (FC) cubical model with the pores equally spaced in the corners of a cube and a pore in the centers of each face.

The pore concentration in the faced centered model is higher than the one in the volume centered arrangement. This leads to the assumption that there will be some differences in the heat flux path between the two arrangements, which would lead to different effective thermal conductivities for the two cases. Table 1 shows the differences in the effective thermal conductivity for the two cases.

**Table 1:** FEM results for face and volume centered models

Porosity [%]	Pores arrangement		Difference [%]	Notes
	Volume centered (VC)	Face centered (FC)		
40.7	0.522	0.5111	2.1	Closed Cells
75	0.197	0.163	17.3	Open Cells
85	0.101	0.078	22.7	Open Cells

As one can see from the values above, the difference between the effective thermal conductivity of the foam with VC pore arrangement and the conductivity of the foam with FC arrangement is small for closed cell foams.

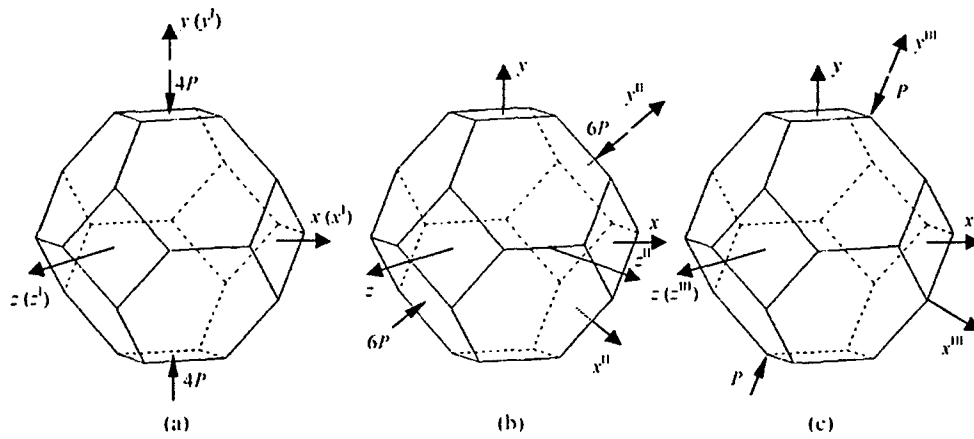
## **1.20 Task (B): Micromechanical Modeling of Carbon Foam**

### **1.21 Theory for Mechanical Model**

As shown in Figure-1, the microstructure of AFRL carbon foam has a three-dimensional (3D) open-cell topology. As a first approximation, a regular tetrakaidecahedron (see Figure-4) can be adopted as the repeating unit to represent this foam microstructure. Tetrakaidecahedron is known to be the only polyhedron that can pack with identical units to fill space (see Figure-5) and nearly minimize the surface energy (Ref. 8). With all of its vertices connected by struts and each vertex shared by four struts, such a 14-sided polyhedron can be generated by uniformly truncating the six corners of an octahedron and contains eight regular hexagonal faces and six square faces.

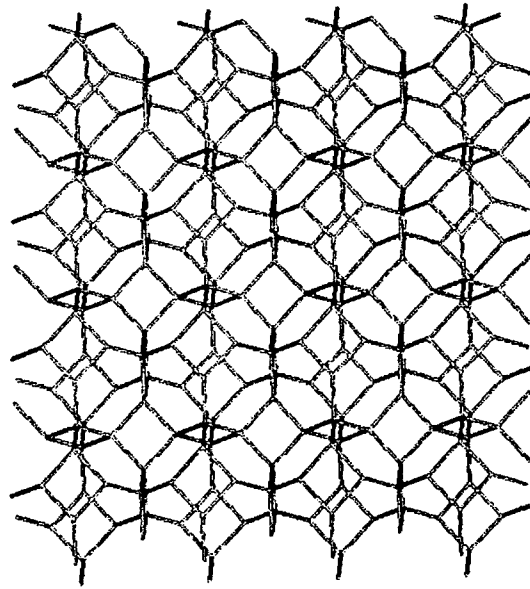
Several micromechanical models for 3D open-cell foams have been developed utilizing tetrakaidecahedral unit cells (see Ref. (9) and references cited therein). Very recently, a micromechanics model for predicting the effective Young's modulus and Poisson's ratio of such foams was provided by Li et al. (Ref. 9) using an energy method based on Castigliano's second theorem, whose predictions compare favorably with those based on available experimental data and finite element analyses. However, only Mode I loading (see Figure-4(a)) was considered there, as was done in almost all previous studies on the topic. Also, how to predict the effective shear modulus was not addressed in (Ref.9). When loads are not applied on two opposite square faces of a tetrakaidecahedral cell, the elastic properties obtained could be different from those determined with the cell subjected to Mode I loading. To illustrate this, two more loading modes, as shown in Figures-4(b) and 4(c), have been considered in the current study. The three loading modes identified here represent three extreme orientations of applied loads. It can be seen from Fig. 3 that the initial length of the cell along the loading direction is the smallest under Mode II loading and the largest under Mode III loading, while the greatest symmetry is present under Mode I loading. For the Mode II and III loading cases anisotropy involved can be significant. Hence, new micromechanical models capable of accounting for the differences in mechanical behavior of foams (cells) under loadings of the three distinct modes are necessitated.

In this project, a new model for predicting the effective Young's moduli, Poisson's ratios and shear moduli of open-cell foams have been developed. The tetrakaidecahedral unit cells illustrated in Figure 4 is adopted, and the matrix method for spatial frames and the homogenization theory for composite materials are employed in the formulation.



**Figure-4:** Tetrakaidecahedral unit cell under loadings of different modes:  
(a) Mode I, (b) Mode II, (c) Mode III.

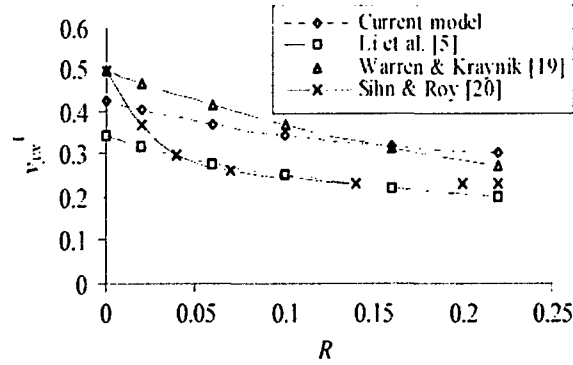
Formulas for predicting the effective Young's moduli, Poisson's ratios and shear moduli have been obtained for open-cell foams under Mode II and Mode III loadings. When the cells in Figure-4 are packed together, the model is represented by Figure 5, shown below.



**Figure-5:** Tetrakaidecahedral cells packed together

To illustrate the new model, a parametric study of sample cases was conducted here for 3D open-cell carbon foams, whose modeling motivated the current study. The MATLAB program (of The Mathworks, Inc.) is used in the computations. Young's modulus ( $E$ ) and Poisson's ratio ( $\nu$ ) of the carbon strut material, which is assumed as isotropic, are, respectively, taken to be 15.61 GPa and 0.33, and the maximum value of the relative foam density ( $R$ ) to be 0.22 (Ref. 9).

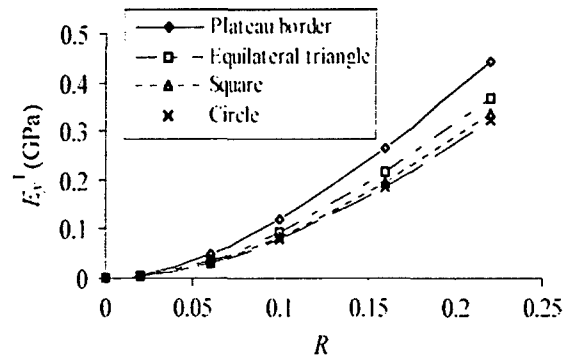
Some the results for shear modulus, Young's modulus and Poisson's ratio in mode I are shown in Figures 6-8. The reader is referred to the publication by Li et al. (Ref. 10) for additional details.



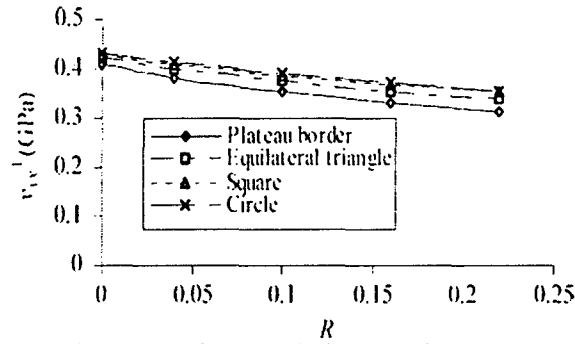
**Figure-6:** Mode I case: shear modulus vs. relative density.

Figure-6 illustrates that, according to the new model,  $\nu_{yx}^I$  decreases monotonically as  $R$  increases. Also, values of  $\nu_{yx}^I$  predicted by the current model are close to 1/3, which is a value suggested by Gibson and Ashby (Ref. 11) based on available experimental data (ranging from 0.15 to 0.4).

The effects of the strut cross-section shape on  $E_y^I$  and  $\nu_{yx}^I$  at various values of  $R$  are shown in Figures 7 and 8 for the Mode I loading case. It is seen from Figure 7 that  $E_y^I$  is the highest for the Plateau border strut cross-section, followed by the equilateral triangle, square and circular strut cross-sections. This descending order conforms to what was observed by Warren and Kraynik (Ref. 12) and Li et al. (Ref. 9). The order of influence of the strut cross-section shape on  $\nu_{yx}^I$  is opposite to that on  $E_y^I$ , as shown in Figure 8. This is in agreement with that noted in (Ref. 9,13).



**Figure-7:** Young's Modulus vs. relative density (Mode I loading)



**Figure-8:** Poisson's ratio vs. relative density (Mode I loading)

For both the Mode II and Mode III loading cases, it is found that the order of influence of the strut cross-section shape on the effective properties of carbon foams remains to be the same as that for the Mode I loading case. For  $R=0.1$ , the maximum relative differences for the three elastic moduli and two Poisson's ratios, resulting from the use of different shapes of the strut cross-section, for the three loading cases are summarized in Table-2. It is noted from Table-2 that the values of the maximum relative differences for all five elastic properties in the three different loading cases are very close except for the Poisson's ratios in the Mode III loading case, which are substantially lower than those in the Modes I and II loading cases.

**Table-2:** Maximum relative differences for elastic properties

	Mode I	Mode II	Mode III
$E_y$	31.47%	33.02%	32.25%
$v_{yx}$	8.93%	8.10%	1.30%
$v_{yz}$	8.93%	7.25%	4.90%
$G_{yx}$	34.05%	33.63%	32.88%
$G_{yz}$	34.05%	33.50%	32.01%

### 1.30 Task (C): Surface modification of carbon Structures

#### 1.31 Rationale for surface modification

Effective reinforcement of carbon structures inside polymer matrix depends primarily on the chemical bonding of the fiber with the matrix material as well as the mechanical interlocking. Sufficient infiltration of the matrix material and thereby even wetting of the fiber material ensures very strong chemical bonding, whereas surface roughness and chirality defines the degree of mechanical interlocking.

However, carbon structures surfaces do not possess good wettability to epoxy matrices. In addition, the interfacial bond strength between the two phases is often inadequate. If the

fiber-matrix adhesion is poor then composite may fail at the interface reducing the strength of the composite (Ref. 14-18). Therefore surface treatment of carbon structures is essential for improving wettability and bond strength, so that they may be used in demanding conditions.

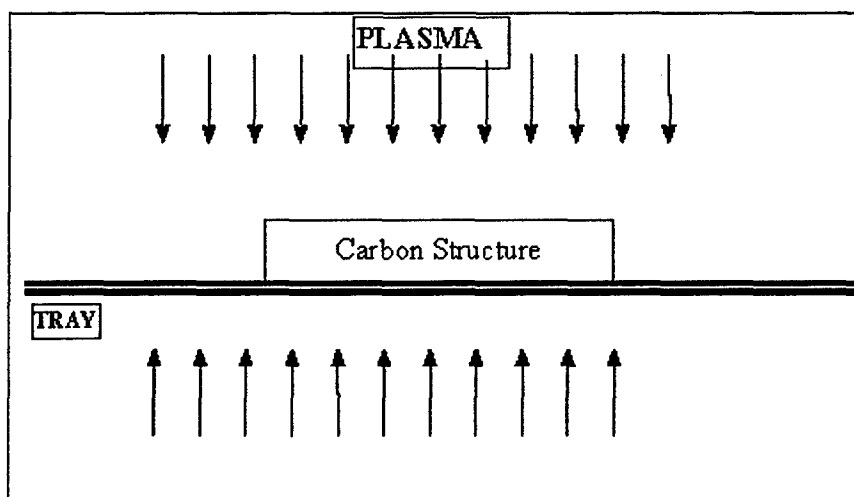
Recently, with the decreasing dimensions of modern composites, various micron/nano size and complex shaped carbon structures are also gaining increased attention. Micro-cellular porous carbon foam and carbon nanofibers are used in this research project. Like all nanomaterials, carbon nanostructures have extremely high surface to volume ratio, which leads to increased chemical reactivity, environmental degradation, tendency to agglomerate, and resistance to uniform dispersion in any matrix.

In this study, plasma assisted oxide thin film deposition techniques were used for effective surface activation of various complex shaped nanoscale carbon core structures. Plasma techniques are well-known for their capability of depositing conformal ultra thin films (several nm) as well as relatively thick films (several  $\mu\text{m}$  thick). By careful selection of the coating chemistry and process parameters, many types of surface modified solids have been obtained over the years using plasma techniques. Applications of plasma films include improved hydrophobicity, hydrophilicity, biocompatibility, printability, adhesion, and corrosion protection (Ref. 19-25).

### 1.32 Experiments

Plasma assisted oxide nano-coatings for surface reactivity is deposited on different carbon structures. This was done using a mixture of HMDSO (hexa-methyl di-siloxane)  $(\text{CH}_3)_3\text{SiOSi}(\text{CH}_3)_3$  and oxygen in a commercial microwave plasma reactor (V15GL manufactured by PlasmaTech Inc.) with multimode microwave source. The aluminum vacuum chamber size is 250mm X 250mm X 250mm (15.6 liters), which uses a microwave generator magnetron of 850 W (electrodeless chamber) and the maximum power available is 600W. The deposition process was performed at 225 W power, 60Pa pressure and ambient temperature. The time was variable for the plasma treatment. Figure-9 shows a typical set-up used for depositing oxide films on different carbon structures.





**Figure-9:** Surface modification in a Plasma Deposition Chamber

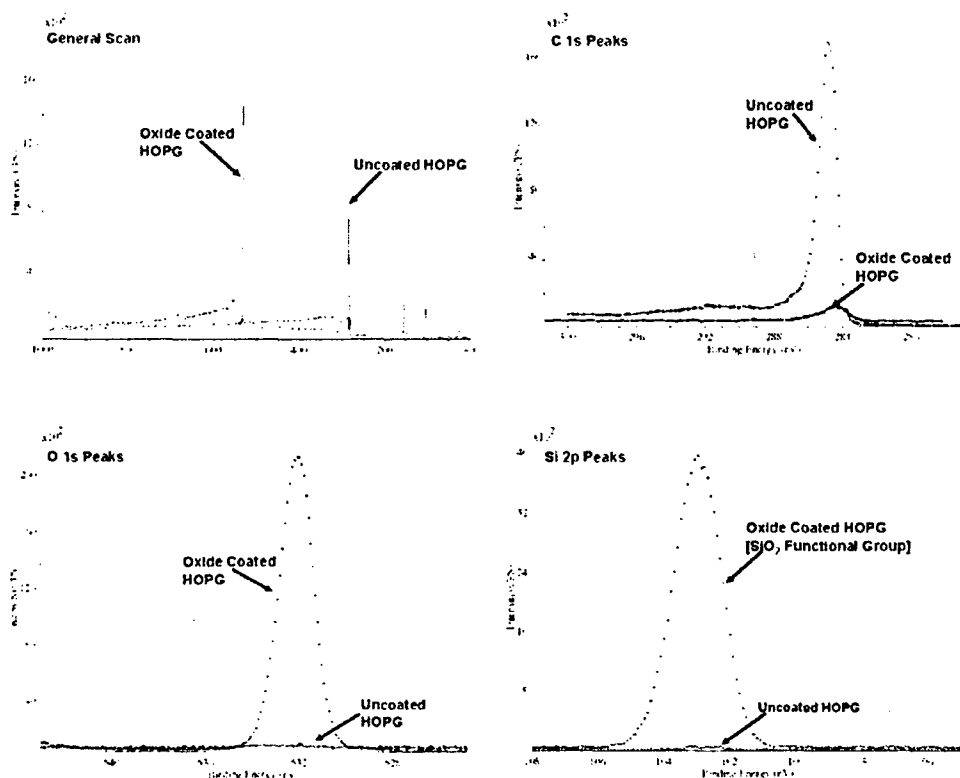
The carbon structures under investigations (microcellular porous foam and nanofiber) are chemically identical to  $sp^2$  graphite (Ref. 26) and therefore, fundamental understanding of growth, coverage and effectiveness of plasma assisted oxide coatings is obtained by depositing ultra-thin films on flat highly oriented pyrolytic graphite (HOPG). As our understating developed the plasma assisted thin oxide films were successfully applied to microcellular porous carbon foam (approx. 100- 150  $\mu m$  pore diameters) and carbon nanofibers (approx. 100nm diameter).

X-Ray Photoelectron Spectroscopy (XPS) (Kratos-Axis Ultra System) with monochromatized Al  $K_{\alpha}$  photons (1486.6 eV) was used for identifying the chemical composition and bonding states of the top few atomic layers of the surface with 110  $\mu m$  diameter aperture. The nano-scale surface morphology and coverage of films were monitored using Atomic Force Microscopy (AFM) at each stage of deposition. Water contact angle measurement was used as a direct measure of effectiveness of the film. Scanning Electron Microscope (JEOL 35) and Transmission Electron Microscope (JEOL CX10) were also used extensively for investigating different material related properties of surface modified carbon structure infiltrated composites.

### 1.33 Results and Discussion

#### Model Studies on Flat Surface

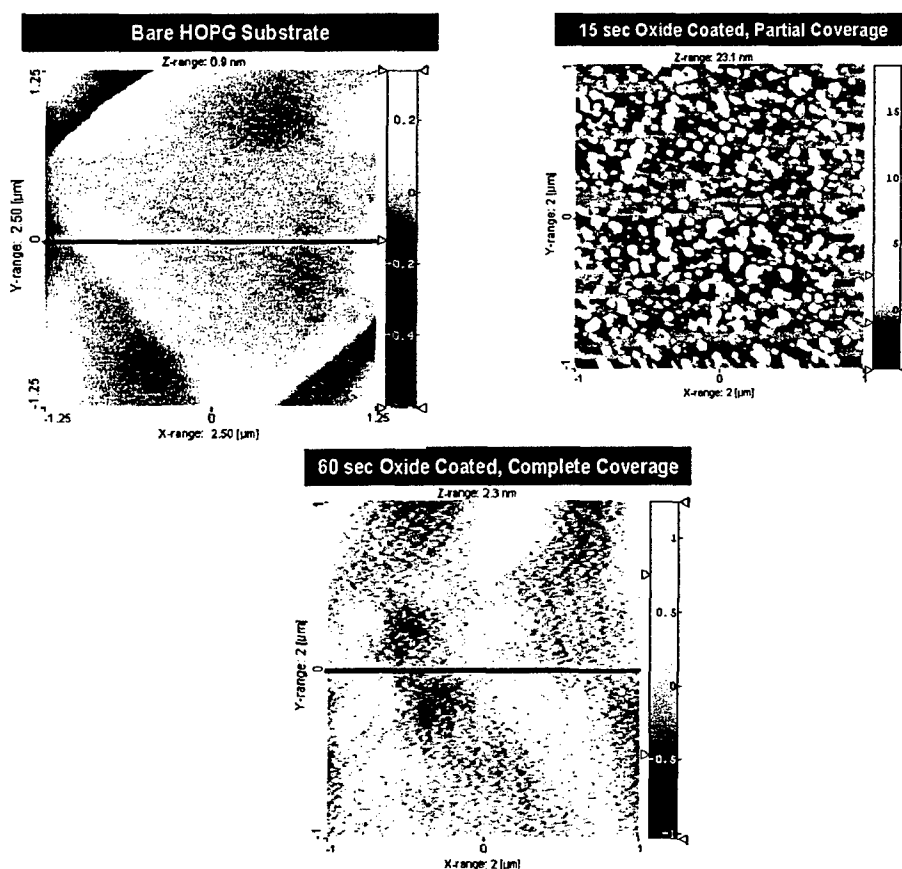
Detailed investigation of the chemical bonding state change as the oxide coating time increases is performed using XPS on highly oriented pyrolytic graphite surface. Figure-10 shows the comparison of XPS scans taken on uncoated and oxide coated HOPG for 4 minutes. It is important to note that the untreated sample contains mainly graphitic carbon (B.E. = 284.75 eV) and minimal oxygen which is basically from ambient atmospheric contaminants. On oxide coated sample, coating deposits contain predominately silicon and oxygen, and minimal presence of carbon. The small carbon peak on the oxide coated surface can be from contamination on the deposited layer due to the delay in transferring the sample from plasma chamber to XPS ultra high vacuum chamber. Figure-4 shows the variation of C 1s, O 1s and Si 2p peak intensities, and their atomic percentages with increasing deposition time. It can be seen, as expected, that the signal from the substrate atoms decrease and those from the coating increases with deposition time. The thickness of this layer can be calculated using the decaying XPS intensity of the substrate peak (Ref. 27-29). Using this data, it could be calculated that the deposition rate was about 4 nm/minute. The fact that no substrate signal is seen after a certain time implies that there is no exposed surface, and that the coating everywhere is significantly thick (at least 7-8 nm) to attenuate all substrate signals.



**Figure-10:** Comparative XPS general and high resolution peaks obtained on uncoated bare HOPG and oxide film coated HOPG indicating diminished substrate peak (graphitic

carbon) and increased coating peaks (oxygen and SiO<sub>2</sub> peaks) after few minutes of coating time.

Tapping mode Atomic Force Microscope (AFM) was used to study the surface morphology of the oxide coating as it grows on HOPG substrates. The AFM topographic images of as received HOPG, and 15 as well as 60 seconds oxide coated HOPG are shown in Figure 11. AFM Surface morphology studies of plasma oxide coatings on HOPG substrate indicates that the substrate was partially covered for 15 seconds coating time and completely covered for 60 seconds coating time, as shown in Figure-5.



**Figure-11:** AFM images of uncoated, partially coated and fully oxide coated HOPG samples.

It can therefore be concluded from the above data that the plasma assisted oxide coatings completely cover the underlying surface within one minutes of coating. The average coating thickness at that time is about 4 nanometers. The next step is to tryout these coatings on more complex carbon structures such as microcellular porous foam and carbon fibers.

### 1.34 Surface Modification of Carbon Foam

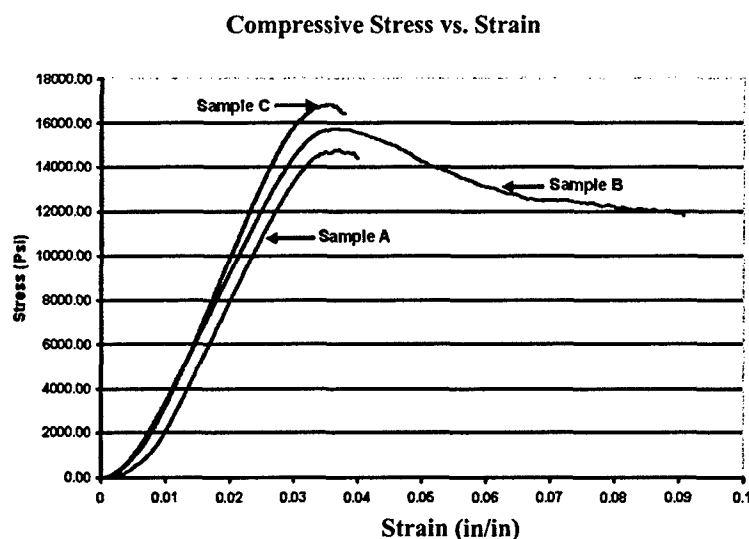
Microcellular carbon foam is a three dimensional porous material that may have future use in lightweight aerospace composites (Ref. 30-32). This structure is known to contain layers, flakes, as well as nano-fibers of graphitic carbon. In order to check if the silica nano-coating enhances the wettability of foam surface with polar fluids (water being an example), carefully weighed foam samples were soaked in water for specific times, and their increase in weight measured as a percentage of original weight. This water uptake capacity was compared between uncoated and oxide coated foams. Table-3 indicates the comparative data, and it is clear that the water uptake is significantly increased due to the oxide coating.

**Table-3:** Comparative increase in the water uptake capacity of microcellular porous carbon foam after plasma assisted oxide coating treatment

Time of Coating (Minutes)	% Wt. Increase of Untreated Carbon Foam	% Wt. Increase of Carbon Foam treated with Oxide plasma
5	32.3	97.5
10	33.0	100.1
15	34.5	102.6
20	47.7	109.1

The oxide coated carbon foam and uncoated bare carbon foam samples were then infiltrated inside the epoxy resins and block compression testing was performed in order to observe the increase in the mechanical properties of the oxide coated foam composites. Detailed SEM analysis was performed on the samples after the compression test to investigate the failure sources and thereby to investigate the bonding enhancement due to nano-scale oxide coating.

Figure-12 shows the compressive test results of the uncoated and coated carbon foam infiltrated in epoxy. It is seen that the plasma oxide coating increases the failure strain of the composite by 125 % compared to uncoated sample.



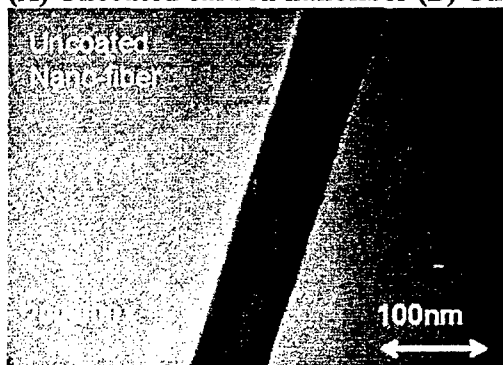
**Figure-12:** Compressive test results of uncoated and oxide coated foam infiltrated with epoxy showing higher compressive strength could be achieved with oxide coating.

### 1.35 Surface Modification of Carbon Nano-fibers

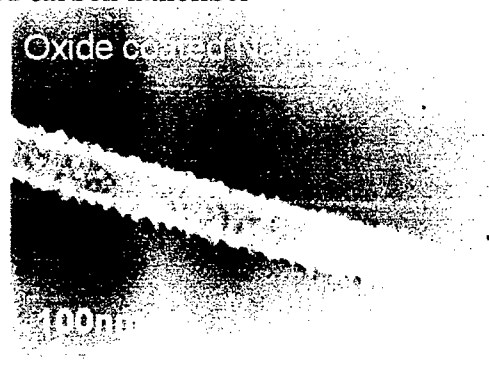
After observing that plasma assisted nano-scale oxide coatings can effectively surface modify the microcellular porous carbon foam structures, similar coatings were applied to vapor grown carbon fibers having diameters in the 100-200nm range.

The TEM image of an individual nanofiber before and after plasma oxide coating is shown in Figure-13 (a) and (b). An ultra thin uniform silica layer can be seen on the oxide coated nanofibers. Detailed TEM observations illustrate that the nanofibers are uniformly coated with silica layer. The thickness of the nano-film is ~4-6 nm covering the entire outer nanofiber surface. When dispersed in an organic solvent such as nitrocellulose, the oxide coated fibers showed significantly lower polymer beading compared to untreated fibers. This observation results from improved wetting leading to a more uniformly dispersed matrix phase. Figure-14 illustrates this phenomenon very well.

**(A) Uncoated carbon nanofiber (B) Oxide coated carbon nanofiber**

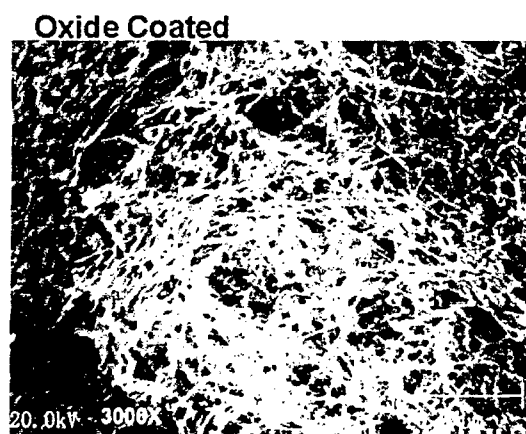
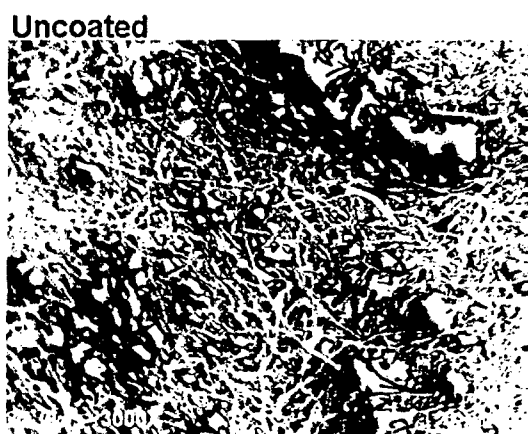


**(a) Uncoated**



**Oxide Coated**

**Figure-13:** High magnification TEM images of uncoated and oxide coated carbon fiber (~200 nm diameter). It is clearly seen that microwave plasma assisted technique can effectively coat complex shaped nano-structured materials



**Figure-14:** SEM images indicating dispersion of uncoated and oxide coated carbon nanofibers in polymeric medium. Enhanced dispersion is achieved with plasma assisted oxide coating of fibers

Plasma assisted nano-scale oxide films are a very viable approach for effectively surface modifying the complex shaped nano-scale carbon structures. These structures once modified can be used in multi-phase composites with enhanced mechanical, thermal and electrical properties.

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## **2.0 Objectives**

The objectives of this proposed study are to develop a knowledge base, and a technology base for carbon foams by analytical, numerical and experimental methods. The models will attempt to capture important features of carbon foams, such as non-isotropic conductivity and randomness in the microstructure. The experimental study will establish reliable techniques for thermal characterization and surface engineering. The joint effort will develop fundamental models to predict the mechanical and thermal properties of graphitic carbon foams and develop surface modifications to tailor the foam properties such as oxidation protection, bonding, and surface flaws and wear resistance.

## **3.0 STATUS OF EFFORT**

Research groups at three different universities have worked to develop fundamental, detailed models of thermal and mechanical behavior of carbon foams, and studied surface modification techniques to enable transition of the foam into several aerospace applications. Three specific tasks were addressed:

- (A) **Thermal Transport in Carbon Foams:** The research team has developed a combined analytical/numerical model to predict thermal conductivity, and have identified, by experimentation, reliable techniques to determine the thermal conductivity of carbon foams. The team used an analytical approach to identify parameters to be calculated by a numerical model.
- (B) **Micromechanical modeling of carbon foam:** The team developed a micromechanics model that includes the effects of cell edges (struts) on the elastic properties of carbon foams; and studied the influence of foam cell size and shape on the macroscopic mechanical behavior of carbon foams using the Voronoi tessellation technique.
- (C) **Coatings for Surface Modification of Carbon Structures:** The researchers have developed surface modification techniques for environmental stability and interface enhancement. The team investigated enhancement of surface wettability, adhesion/bond control, and coatings for extreme applications.

## **4.0 Accomplishments and New Findings**

The research effort has produced a knowledge base for carbon foams that can be used to develop technical applications for this novel material. Carbon foam is a novel micro-cellular material that has potential structural and thermal management applications. These foams have special advantages in multi-functional aerospace applications where a good

combination of light-weight, structural and thermal properties is required. Unlike graphitic carbon fibers, the foam can be processed to be isotropic or anisotropic; therefore, it is a promising candidate for near-net shape fabrication. Thermal management is a strategic application where carbon foam has the most versatility. It can be used as a thermal insulator or as a very good conductor over a wide range of temperatures.

The research has defined and enhanced understanding of the properties of the new carbon foams. Prior to this investigation, very few studies on the properties of carbon foams were reported in literature. The research has provided design tools that make it possible to meet structural, thermal and environmental stability requirements for carbon foam in aerospace applications.

The research findings are summarized in the following three sections, as reported by the three universities. These sections are based on the journal publications of the investigators.

- (A) **Thermal Transport in Carbon Foams:** M.K. Alam (Principal Investigator)  
A finite element model has been used to determine the thermal conductivity of high porosity foams with anisotropic strut-node structure. The foam model was constructed by solid modeling software using interconnected spherical pores. The thermal conductivity result was obtained in the form of a non-dimensional effective thermal conductivity.

The results show that semi-empirical constants can only be valid for specific foams and porosity ranges. The results also show that the anisotropy of the carbon foams, mainly the ratio between the node and ligament thermal conductivities, significantly affects its bulk thermal conductivity. Although the nodes make up most of the mass, the bulk conductivity is not dominated by the node conductivity.

- (B) **Micromechanical modeling of carbon foams:** X.-L. Gao (Principal Investigator)  
A micromechanics model for three-dimensional (3D) open-cell foams has been developed by using the matrix method for spatial frames and tetrakaidecahedral unit cells. The effective elastic properties of foams are determined utilizing unit cells subjected to loadings of three different modes. The newly obtained member stiffness submatrices for a 3D frame member undergoing simultaneous axial, transverse shearing, flexural and torsional deformations and having a circular or non-circular cross-section are employed. The use of these stiffness submatrices enables the incorporation of all four deformation mechanisms (i.e., stretching, shearing, bending and twisting) possible under the specified loadings and the treatment of four different strut cross-section shapes (i.e., circle, square, equilateral triangle and Plateau border) in a unified manner. The formulas for determining the effective Young's moduli, Poisson's ratios and shear moduli of open-cell foams are derived using the composite homogenization theory. The new

formulas, in which the cell displacements are obtained employing the matrix method, include all necessary microstructural and material parameters, unlike those provided by existing models. It is shown that the foam elastic properties depend on the relative foam density, the shape and size of the strut cross-section, and the Young's modulus and Poisson's ratio of the strut material. By applying the new model, a parametric study of sample cases involving the three loading modes and the four strut cross-section shapes is conducted for carbon foams, whose modeling motivated this work. The predicted values of the effective properties agree favorably with those based on existing models and experimental data for the Mode I loading case, which is the only case that has been well studied. For the Mode II and Mode III loading cases, the formulas and the subsequent sample results are presented in a systematic manner for the first time. Comparisons of the predicted effective elastic properties for the three loading cases quantitatively show that carbon foams exhibit certain degrees of anisotropy. q 2004 Elsevier Ltd. All rights reserved.

**(C) Coatings for Surface Modification of Carbon Structures, S. M. Mukhopadhyay (Principal Investigator)**

Carbon core structures are an important class of reinforcing materials for many composites, and modification of their surfaces is essential for suitable wettability and bonding with the matrix material. Enhanced wettability is required for better dispersion and prevention of voids, and appropriate bond strength with matrix material is desired for obtaining the necessary mechanical, electrical and thermal properties. In nanocomposites, these carbon core structures themselves are of <100nm dimensions, therefore the coating thickness has to be substantially smaller (few nanometers), yet be durable and effective. In this task, microwave plasma assisted oxide coatings having thickness < 5nm has been successfully be applied to such complex shaped nano-structured carbon materials. As the first step, fundamental issues of growth, coverage and functionality of these nano-scale oxides have been investigated on flat model surfaces. These coatings were found to completely cover the underlying surface and form nano-layers of SiO<sub>2</sub> type compound, which increases the surface reactivity. Subsequently, these coatings were successfully applied to micro-cellular porous carbon foam and carbon nano-fibers. It was seen that they are indeed in enhancing wettability and bond strength of these porous and uneven structures.

## 5.0 Personnel Supported:

### A. Ohio University

Dr. M. K. Alam (PI), Professor  
Adriana Druma (Graduate Student)

### B. Michigan Technological University/Texas A & M University

Dr. Xin-Lin Gao (PI), Professor  
Ke Li, (Graduate Student)

### C. Wright State University

Dr. Sharmilal Mukhopadhyay (PI), Professor  
Rajashekhar V. Pulikollu (Graduate Student)

## 6.0 Journal Publications

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## 7.0 Interactions/Transitions

### 7.1 Presentations

1. A.M. Druma, M.K. Alam, R. Watts, M. Kistner, "Thermal Conductivity and Structural Properties of Emerging Composite Materials," *Proceedings of the SAMPE 2004 Symposium and Exhibition*, Long Beach, CA, May 16-20, (2004).
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## **7.2 Consultative Functions**

None

## **7.3 Transitions**

None

## **8.0 New Discoveries, Inventions, or Patent Disclosures**

None

## **9.0 Honors/Awards**

White Research Award presented to Dr. M. K. Alam by Ohio University in 2004.